

FORM PTO 1390 (REV 11-2000)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE	ATTORNEY'S DOCKET NUMBER H0610.0000
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371			U.S. APPLICATION NO. (if known, see 37 CFR 1.5) 10/019485
INTERNATIONAL APPLICATION NO. PCT/EP00/05275	INTERNATIONAL FILING DATES June 7, 2000	PRIORITY DATE CLAIMED July 1, 1999	
TITLE OF INVENTION CONTINUOUS DEHYDRATION OF ALCOHOL TO ETHER AND WATER USED AS FUEL FOR DIESEL ENGINES			
APPLICANT(S) FOR DO/EO/US Svend-Erik Mikkelsen, et al.			
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:			
<ol style="list-style-type: none"> 1. <input checked="" type="checkbox"/> This is a FIRST submission of items concerning a filing under 35 U.S.C. 371. 2. <input type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing 35 U.S.C. 371 3. <input checked="" type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371 (f)). The submission must include items (5), (6), (9) and (21) indicated below. 4. <input checked="" type="checkbox"/> The US has been elected by the expiration of 19 months from the priority date (PCT Article 31). 5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371 (c)(2)) <ol style="list-style-type: none"> a. <input checked="" type="checkbox"/> is attached hereto (required only if not communicated by the International Bureau). b. <input checked="" type="checkbox"/> has been communicated by the International Bureau. c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US). 6. <input type="checkbox"/> An English language translation of the International Application as filed (35 U.S.C. 371 (c)(2)). <ol style="list-style-type: none"> a. <input type="checkbox"/> is attached hereto. b. <input type="checkbox"/> has been previously submitted under 35 U.S.C. 154(d)(4). 7. <input type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3)) <ol style="list-style-type: none"> a. <input type="checkbox"/> are attached hereto (required only if not communicated by the International Bureau). b. <input type="checkbox"/> have been communicated by the International Bureau. c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired. d. <input type="checkbox"/> have not been made and will not be made. 8. <input type="checkbox"/> An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)). 9. <input type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)). 10. <input type="checkbox"/> An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)). 			
Items 11 to 20 below concern document(s) or information included:			
<ol style="list-style-type: none"> 11. <input type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98. 12. <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included. 13. <input checked="" type="checkbox"/> A FIRST preliminary amendment. 14. <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment. 15. <input type="checkbox"/> A substitute specification. 16. <input type="checkbox"/> A change of power of attorney and/or address letter. 17. <input type="checkbox"/> A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825. 18. <input type="checkbox"/> A second copy of the published international application under 35 U.S.C. 154(d)(4). 19. <input type="checkbox"/> A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4). 20. <input type="checkbox"/> Other items or information: 			

U.S. APPLICATION NO. (if known, see 37 CFR 1.53) <div style="font-size: 1.5em; font-weight: bold;">10/019485</div>	INTERNATIONAL APPLICATION NO. PCT/EP00/05275	ATTORNEY'S DOCKET NUMBER H0610.0000
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21. ☒ The following fees are submitted:

BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)):

☐ Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO\$1000.00

☒ International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO\$890.00

☐ International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO\$710.00

☐ International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4)\$690.00

☐ International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4)\$100.00

ENTER APPROPRIATE BASIC FEE AMOUNT =

Surcharge of \$ _____ for furnishing the oath or declaration later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492 (e)).

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	
Total claims	5 -20 =		x \$18.00	\$
Independent claims	1 -3 =		x \$84.00	\$
MULTIPLE DEPENDENT CLAIM(s) (if applicable)				\$
TOTAL OF ABOVE CALCULATIONS =				\$
<input type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2.				\$
SUBTOTAL =				\$ 890.00
Processing fee of \$ _____ for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492 (f)). +				\$
TOTAL NATIONAL FEE =				\$ 890.00
Fee for recording the enclosed assignment (37 CFR 1.21 (h)). Assignment must be accompanied by appropriate cover sheet (37 CFR 3.28, 3.31) (_____ per property). +				\$
TOTAL FEES ENCLOSED =				\$ 890.00
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a. ☒ A check in the amount of \$ 890.00 to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. _____ in the amount of \$ _____ to cover the above fees. A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required or credit any overpayment to my Deposit Account No. 04-1073 . A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

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Att'y Docket No.: H0610.0000
(PATENT)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:
Svend-Erik Mikkelsen et al.

International Appln. No.:
PCT/EP00/05275

Application No.: Not Yet Assigned

International Filing Date:
June 7, 2000

Filed: Herewith

For: CONTINUOUS DEHYDRATION OF
ALCOHOL TO ETHER AND WATER
USED AS FUEL FOR DIESEL ENGINES

PRELIMINARY AMENDMENT

Commissioner for Patents
Washington, DC 20231

Dear Sir:

Preliminary to examination, please amend the above-referenced application as follows:

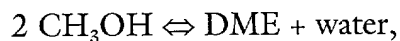
IN THE CLAIMS:

Cancel claims 1-6, without prejudice.

Add new claims 7-11 as follows:

7. (New) A process of preparing an oxygenated diesel fuel composition sustainable for use in compression ignition internal combustion engines, comprising methanol, dimethyl ether and water, comprising the step of converting methanol containing up to 20% w/w of water and up to 20% w/w of ethanol or higher alcohol in a

catalytic dehydration reaction, the methanol being converted to dimethyl ether according to the reaction scheme:



using a catalytic converter on board of a vehicle, wherein the dehydration temperature is between 200°C and 450°C and wherein the pressure is between 10 and 400 bar.

8. (New) A method of operating a compression ignition engine on a fuel obtainable by the process of claim 7, by injecting the fuel into the combustion chamber of the engine and combusting the fuel with air, wherein the concentration of methanol is between 5 and 50% w/w and wherein the air for combustion is preheated to a temperature of at least 60°C.

9. (New) The method of claim 8, wherein the combustion air is preheated to a temperature of at least 100°C.

10. (New) The method of claim 8, wherein the combustion air is preheated by exchange with the exhaust gas.

11. (New) The method of claim 8 applied to vehicles, ships, trains or in stationary diesel engines for power and heat supply.

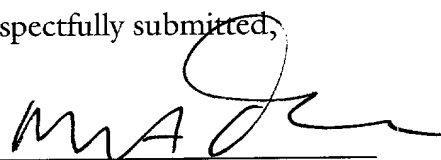
REMARKS

New claims 7-11 are substituted for original claims 1-6 to reduce the filing fee and for other reasons. Applicants reserve the right to pursue the original claims and other claims in this application and in other applications. Favorable action on the application is solicited.

Dated: December 31, 2001

Respectfully submitted,

By



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Continuous Dehydration of Alcohol to Ether and Water
used as Fuel for Diesel Engines

FIELD OF INVENTION

This invention relates to improved fuels for compression ignition internal combustion engines equipped with inlet air heater. More particular, this invention relates to oxygenated diesel fuel compositions, which contain a combination of alcohol, ether and water. The ratio of alcohol/ether/water is selected by catalytic dehydration of alcohol. The fuel compositions are obtained by means of a catalytic dehydration unit by which a catalytic dehydration unit by which heat is provided by the engine exhaust provides heat by the engine exhaust. The obtained advantages are low emissions and high engine efficiency.

BACKGROUND OF THE INVENTION

Since Rudolf Diesel invented the basic diesel engine concept in 1893 constant improvements have been reached. The engine efficiency has increased from 5 to more than 40% today. Through decades of continues development work and efforts, reliability, drive ability and performance of this engine type have resulted in global acceptance and huge mass production. The number of diesel engines employed in vehicles, ships and power stations world wide have established a need for constant increased supply of fuel despite of all the above mentioned obtained engine improvements. The good fuel economy is one of the most important reasons for the achieved success measured as number of engines produced and sold compared to the diesel engines counterpart the spark ignition engine or Otto-cycle engine. Depending on engine size, fuel economy in diesel engines today is in

the area of 20-40% more efficient than Otto-engines. Associated with the more and more wide spread use of internal combustion engines operated on fossil fuel, ecological and environmentally problems have aroused. Concerns for human health and global environmental changes due to the gases and solid particular matter associated with fuel combustion are of increasing international attention and concern. More specific, the amount of particulate matter, oxides of nitrogen and sulphur, is a problem for even modern diesel engines. Non-combusted fuel and carbon monoxide pose another negative environmental and human influence. To reduce those harmful emissions, improvements of various areas are under constant development. Among these main areas are: fuel injection systems, combustion chamber designs, turbocharger design, heat management systems like internal gas coolers, exhaust gas recycle, etc. Externally catalyst for various applications, absorber systems and particulate trap devices are used and improved. The quality of the diesel fuel is another way to improve combustion and reduce emissions. Better exhaust quality is obtained, to mention some, by desulphurisation, hydrogenation and by controlling fuel distillation properties at the refining process.

In spite of all the achieved improvements, the overall air quality still poses a huge problem on earth due to the number of vehicles running. The present fuel consumption and steady increase causes still more concerns for lack of sustainability for future energy supply. There are obviously three main problems to attend to:

- Future energy supply security
- Air quality in urban areas (toxic components)
- Global air changes (greenhouse gases)

Carbondioxide is a greenhouse gas believed to increase global atmospheric temperature. Limitations of the amount of carbondioxide produced and emitted associated with combustion in internal combustion engines are closely linked to the efficiency of the engine. Generally speaking, diesel engines operate with 30% better fuel efficiency compared to spark assisted Otto-engines. The 30% better fuel efficiency corresponds roughly to 30% less emitted carbondioxide. Natural gas and methanol are both Otto-engine fuels. In the above description a method to improve efficiency for methanol combustion in internal combustion engines by reformulation on board a vehicle, gaining diesel efficiency is outlined.

Generating methanol from natural gas is today an established technology worldwide. More than 25 MMT is produced annually. The methanol production capacity has increased every year for more than a decade now. Known reserves of natural gas recourses, relevant for further production and expansion counts for at least an equivalent energy mass compared to proven oil reserves. While oil and oil deviated fossil fuels are the main source of energy used for transportation today, natural gas and natural gas deviated products counts for a limited use, directly as compressed natural gas (CNG) and liquefied natural gas (LNG) or indirectly in forms of and liquefied deviated products like methanol, ether and Fischer Trops (FT) type products. One of the possible strategies for energy production is more efficient use of the recourses. Another coherent possibility is to change the ratio between oil and gas deviated products used towards the gas technologies. A change in the direction of more efficient utilisation of natural gas based processes

combined with more efficient utilisation of the generated fuels used in the transportation sector is a key issue in the present invention. Increasing efficiency could be achieved by increasing production capacity of new industrial production plants (economy of scale) as an example. This combined with development of high efficient use of the produced deviates makes the overall efficiency increase possible. More specifically methanol is today used as a fuel in Otto-engines directly or mixed with gasoline neat or chemically reacted as MTBE as octane improves. In all cases the fuels are suited for Otto engines and used in Otto engines. If the produced fuel could be used directly or indirectly in a diesel cycle engine, increased fuel efficiency of 20-40% would be expected. Many ways have been suggested to achieve this.

To mention one close related way, it has been suggested to convert methanol to dimethylether (DME) and use this as a diesel fuel (US Patent No. 5,906,664). DME can also be produced in a direct process (Topsoe patent DK 171,707) and save both investments in plant construction and also in operating costs. DME has shown to be a superior diesel fuel with good combustion characteristic. No particulate is formed and lower emissions of NOx are achieved with no penalty in fuel efficiency. Fuel efficiency is corresponding to diesel fuel based upon energy equivalent mass. However, infrastructure and logistic are more expensive than traditional requirements for gasoline and diesel.

In order to achieve the good efficiency of DME and to avoid the more expensive logistic, methanol is produced, distrib-

uted and used as the basic energy carrier. Then methanol is converted on board a vehicle to DME. Methanol conversion is performed by means of a catalytic converter. Catalyst used for the dehydration process is known and applied for in the aerosol industry. The catalytic converter is heated by surplus heat from the exhaust gas using a heat exchanger. The obtained fuel is suitable as fuel for direct and indirect diesel injection engines provided with a heater on the air intake.

Storing a small amount of the generated fuel makes instant engine start possible. Further advantages using the generated fuel composition are the CO-produced water. This water reduces formation of oxides of nitrogen (NOx) due to a reduction in combustion temperature to even lower level than operation with neat DME. Small amounts of unconverted methanol, which is present due to thermodynamic reasons, is acting as CO-solvent for the DME/water mixtures securing that only one single phase of stable fuel is obtained with a very low freezing point.

The present invention relates to a method by which the goal of high efficiency and low emissions can be reached by modifying an Otto-fuel with low fuel efficiency to a diesel fuel with high fuel efficiency. More specifically, methanol, which is a fuel for Otto-engine with high Octane number (115) and low Cetane number (5), is converted to preliminarily dimethylether (DME) with low Octane number and high Cetane number (>55) and water by a catalytic dehydration process. The obtained fuel comprises some unconverted methanol, generated ether as the main component and associated water. This fuel composition is used directly as fuel

in a diesel engine. The generated dimethylether has a high Cetane number (>55) and is known to be a superior diesel fuel. From two mole of methanol, one mole of DME and one mole of water are formed. The chemical reaction can be written as follows:

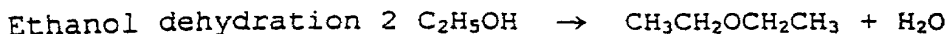


Equilibrium limitations prevent a 100% conversion of methanol to DME and water. This means that methanol conversion or dehydration to ether and water always leaves some unconverted methanol unless a separation or distillation process is applied. However, experimental engine test has concluded that separation of the produced fuel is not required. It has been found that only preheating of the inlet air combined with the catalytic dehydration process is necessary to obtain smooth combustion in a diesel engine.

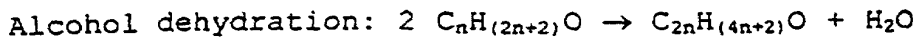
The fuel composition obtained by dehydration of dry methanol then consists of a constant molecular ratio between DME and water, which is 1:1. Expressed as mass ratio, the ratio of DME and water will be 2.56. Depending on methanol dehydration degree the amount of methanol in the DME/water produced will counts for 0 at 100%. 0% at 100% conversion (theoretically) and 100% at zero conversion. This explanation is shown graphically as a trapeze diagram in Fig. 1 attached.

Modern industrial methanol production plants today are equipped with high quality distillation utilities in order to make high quality chemical grade methanol. If a more simple separation is applied, savings in investment and op-

erating can be obtained. A cheaper fuel grade methanol is then obtained. With the trade-off that this crude or technical grade methanol contains some ethanol, higher alcohol and water up to 20 wt%. However, these alcohol are dehydrated as well as methanol is dehydrated yielding good diesel quality property. The chemical reaction for generating diethylether from ethanol can be written as follows:



The formed diethylether has a low self-ignition temperature (160°C), which is even lower than DME (237°C). Higher alcohol is dehydrated likewise to ethers and water. The molecular ratio between ether and water formed are always 1:1 increasing molecular weight of the ether increases the weight ratio between the ether and the water. Methanol dehydration results in a mass ratio between formed dimethylether and water corresponding to 2.6. Ethanol dehydration yields a mass ratio of 4.1 between formed diethylether and water. Evaluation of test fuel containing diethylether is given in Table 6 described later. Minor concentrations of higher alcohol are converted to ether according to the above-described chemical equation.



To evaluate the influence of water, experimental studies have confirmed that very high concentrations can be tolerated on the condition that sufficient high air inlet temperature are available according to the present invention. Typically, water is closely associated with alcohol production. The price for separation of the alcohol from the wa-

ter in mixtures depends upon the specific production unit. Usually, this separation cost raise exponentially with the demands for purity. A trade-off in this situation with the fuel price, the price for moving around with the fuel containing water and the fuel efficiency obtained in the engine have to be determined in all situations. Generally speaking more water can be accepted produced and used close to the engine.

Detailed results of experimental evaluation on aqueous fuels are given in Table 7 described later.

SUMMARY OF THE INVENTION

The present invention relates to a more sustainable energy supply situation for the transportation sector for the coming decades if used as the invention suggests. Higher overall fuel efficiency is obtained. At the same time, reduced emissions of environmentally harmful components such as particulate matter (PM) and oxides of nitrogen (NOx) are achieved. Also a reduction in carbondioxide (CO₂) is obtained. As a consequence of the technological achievements written in the present invention, a ramification of the possible sources for fuel manufacturing are enabled; not only neat methanol produced from natural gas, also methanol produce from coal or renewable like wood can be used. Even use of garbage could be possible. Higher alcohol especially ethanol can be produced from renewable sources and used in blends with methanol. Mixtures containing methanol/ethanol and water are suitable within a specified written limited area. The alcohol mixtures are converted on board a vehicle to the associated ether and water. The obtained fuel compo-

sitions are suitable for use in compression ignition internal combustion engines equipped with inlet air heater. Use of diesel in diesel engines is associated with a surplus of heat released during combustion. Exhaust gas temperatures from 200°C to more than 500°C are typical depending on the engine load. Most of this energy contained in the exhaust gas is normally wasted as heat loss. In the present invention this energy is used for the evaporation and heating of the alcohol fuel used. Further, the surplus heat is used to preheat the air used for the combustion.

The synergetic combination of fuel and heat management results in the achieved high fuel efficiency and the low concentrations of harmful spices in the tail gas.

Experiments on a small single cylinder Yanmar YDG 3000 diesel engine equipped with external air heating have confirmed the fuel composition ability to ignite without any glow or spark assistance with the specified advantages.

BRIEF DESCRIPTION OF THE DRAWINGS AND FIGURES

Fig. 1 is a trapeze diagram showing physical conditions for DME, methanol and water, equilibrium conditions, tested fuel compositions and various patented areas.

Fig. 2 is a simplified flow sheet for on board alcohol conversion.

Table 1: Energy content in tested fuels.

Table 2: Test fuel compositions.

Table 3: Test fuel No. 1 - DME as reference fuel.

Table 4: Test fuel No. 2 - Diesel as reference fuel.

Table 5: Test fuel No. 3 -

DME/methanol/water=60/15/25, wt/wt%.

Table 6: Test fuel No. 4 - DME/methanol/water=60/20/20,
wt/wt%.

Table 7: Test fuel No. 5 -

DME/methanol/water/DEE=50/20/20/10, wt/wt%.

Table 8: Test fuel No. 6 - DME/methanol/water=48/4/48
wt/wt%.

Table 9: Summary of achieved results.

DESCRIPTION OF THE INVENTION

The system shown in Fig. 2 consist of a methanol (alcohol) storage tank A. Methanol is pumped by means of pump 1 from the storage tank through B, which is a feed/effluent heat exchanger, where methanol is evaporated and heated. C is a start-up heat exchanger. D is the catalytic dehydration reactor converting the alcohol to ether and water. Reacted methanol leave D through the feed effluent heat exchanger as equilibrated methanol, DME and water. Pressure in the D is adjusted by means of the backpressure valve V1. Through E the fuel is cooled and condensed. In F the fuel is collected. For system start-up, F is supplied with fuel from external source. For normal operation the content in F is sufficient for engine start-up. A level control in F is electronically managing the required fuel production in D. From F the fuel is pumped with pump 2 to the common rail G. The fuel is by electronically controlled injectors injected directly into the cylinders of the engine H. Surplus fuel from the cylinders are returned to F. In order to keep sufficient fuel pressure in G, some surplus fuel is recycled to F through V2, the control valve for system pressure in

G. Exhaust air is passed a turbo charger I. Heat from exhaust gas is exchanged and transferred to I into the inlet air used in the diesel engine. J is a separate start up heater. V3 is a back pressure valve connected to I. The dehydration reactor D is heated by exhaust gas from I.

Through L, a shunt valve the needed amount of heat is sent to D. Excess heat is sent to the catalytic oxidation catalyst N. C and M is a start up heaters. Exhaust gas passing D is further passed through the catalytic oxidation catalyst N. In N the main part of CO and HC is converted to CO₂ and water. From N the stream is led to the open air. Verifying the given description accomplished with detailed experimental documentation a test stand with single cylinder diesel engine and laboratory equipment for preparation fuel mixtures were build. Results are accordingly given below.

EXPERIMENTAL SET-UP

Preparation of Test Fuels

In order to determine the specific ignition quality of the diesel fuel, compositions mentioned a dedicated test engine and associated fuel supply system was set up according to the following description.

Fuel mixtures were prepared in 1 kg batches stored in a 2 litres pressure cylinder. To determine the specific amount of each component, the cylinder was placed on a balance. First DME was added and the scale read out noted. Then a pre-mixed composition of methanol and water was entered another sample cylinder and the amount noted. This mixture was pressurised to 10 bar g with nitrogen. Both cylinders were equipped with valves in top and bottom. The bottom

valves of the cylinders were connected by a PTFE hose. By opening both valves, the methanol/water mixture was transferred to the DME containing cylinder driven by the difference in pressure and the valves were closed. The balance read out was noted again. The final composition in wt/wt% was then calculated.

Fuel Supply and Test Engine Set-Up

Now the PTFE hose was connected to a three-way valve inlet position one. Inlet position two was connected to a neat DME supply pressurised by a pump to 18 bar. The outlet of the 3-way valve was then connected to the fuel pump on a single cylinder commercial standard four stroke diesel engine of the type Yanmar model YDG 3000. By changing the direction of the three-way valve toggling between neat liquid DME and test mixture was enabled.

Engine cylinder volume was 0.273 litres. Injector opening pressure was adjusted to 100 bar g and the excess fuel from the injector was recycled to the fuel pump inlet. The 3-way valve was set to position two having neat DME connected to the engine. The engine was now ready for start.

Inlet Air Heater

An external electrical heater on the inlet air powered by external electricity, was build. A thermostat regulator was adjusted to heat, the inlet air to a selected temperature between ambient and 150°C. Thermocouples mounted on the inlet and the outlet exhaust of the engine were connected to a temperature indicator.

Exhaust Gas Analysis

A part stream of the exhaust gas from the engine was pumped through an analytical system. Quantitative on-line determination of carbon monoxide (CO), hydrocarbons (HC), oxides of nitrogen NO_x (NO+NO₂), carbon dioxide (CO₂) and oxygen (O₂) were enabled. For the experimental purpose, no catalytic oxidation catalyst was used. This was chosen in order to have the best possible impression of the combustion quality. Equipment measuring the revolutions of the engine was also installed.

Engine Load

Load on the diesel engine was established by connection an electrical heater to the applied 220 Volt electricity generator. A load of 1-2 kW corresponding to 40-80% of nominal maximum capacity was established.

Fuel Consumption

The fuel consumption was determined by logging the read out from the balance carrying the cylinder with the test fuel mixture every minute during the tests. Calculation of the specific fuel consumption was based on the energy content given in Table 1. In the calculations no corrections for the energy used for the external inlet air heating were used. The calculated fuel consumption for the different fuels tested was all performed and compared at the same engine load (1kW). Below a detailed description of fuel consumption and emissions obtained at various air inlet temperatures given.

Table 1
Fuel properties

Fuel component	Lower Heating Value MJ/Kg	Molecular weight
Diesel (MK-1)	42.5	-
DME	28.84	46.07
DEE	34.3	74.13
Methanol	19.7	32.04
Ethanol	26.8	46.07
Water	0.0	18.01

Testing of Reference and Diesel Fuel Mixtures

According to Table 2 various test fuels have been prepared to investigate combustion characteristics, exhaust gas compositions and also determine the fuel efficiency.

Table 2
Fuel compositions

Test fuel No.	DME wt %	Methanol wt %	Water wt %	DEE wt %	Diesel wt %
1	>99.5	0	<0.5	0	0
2	0	0	0	0	100
3	60	15	25	0	0
4	60	20	20	0	0
5	50	20	20	10	0
6	48	4	48	0	0

The procedure for testing these various test fuels is outlined below.

EXAMPLES OF THE INVENTION**Example 1****Neat DME as Reference Fuel (Fuel No. 1)**

The engine was started on neat DME. The engine load was adjusted to 1 kW and stabilised. The engine was operated for approximately 5 minutes - until stable exhaust temperature was seen. All measurable parameters as described were measured and noted every minute on a log sheet. Then the inlet temperature of the intake air was heated to 125°C. After stabilisation a new set of measurements was executed. Inlet air heating was cut-off and the engine was stopped.

The results of these two data sets are shown in Table 3. All measured components. (NOx, HC, CO, O₂, CO₂) in the exhaust gas are given as volume amounts. Fuel consumption (FC) are given as actual consumption in g/hour and as calculated values as MJ/hour based on energy content specified as lower heating value (LHV) of the specific fuel tested given in Table 1.

Table 3

Inlet Air temperature °C	Exhaust temperature °C	FC gram/h	FC MJ/h	NOx PPM	HC PPM	CO %	O ₂ %	CO ₂ %
10	206	788	22.74	119	163	0.073	16.1	3.4
124	301	756	21.80	259	64	0.042	14.3	4.6

Discussion of Results

It was seen that increased inlet temperature increased the fuel efficiency at the specified load. Further, it could be

concluded that emissions of HC and CO were reduced with a trade off on the emissions of NOx. NOx emissions were almost doubled as a consequence of the increased inlet temperature.

Example 2

Diesel as Reference Fuel (Fuel No. 2)

In order to compare fuel consumption and emissions diesel fuel was applied as fuel. The Swedish low sulphur fuel type MK-1 was selected for this purpose. The engine was operated without preheated air only. The results are given in Table 4.

Table 4

Inlet Air tempera- ture °C	Exhaust tempera- ture °C	FC gram/h	FC MJ/h	NOx PPM	HC PPM	CO %	O ₂ %	CO ₂ %
10	204	538.8	22.63	388	31	0.063	15.7	3.8

Discussion of Results

It appears that the efficiency is almost the same as obtained with neat DME fuel. Emissions of NOx were more than 3 times higher with diesel fuel compared to the DME operation.

Example 3

Diesel Fuel Composition (Fuel No. 3)

DME/methanol/water composition = 60/15/25 wt/wt%.

According to the chemical equilibrium calculations shown in Fig. 1 concentrations close to 60% DME, 15% methanol and 25% water all by mass are obtained at 300°C dehydration temperature and 20 bar. A fuel mixture having this composition was prepared and tested according to the test procedure described above. The diesel engine was started on neat DME. Load on the engine was set to 40% by means of the electrical heater (1Kw). Air inlet was heated to 124°C and stabilised. The 3-way valve was switched from neat DME to fuel No. 3. It was observed that the engine kept running on the specified fuel composition. The fuel consumption and emissions were measured. The air inlet temperature were reduced to 105°C. The engine operation was slightly uneven. Fuel consumption was calculated and emissions measured. Further reduction of air inlet temperature to 80°C resulted in a definitely more uneven operation. A new data set was assembled.

A failure on the exhaust gas thermocouple occurred during this test. Results are given in Table 5 below.

Table 5

Inlet Air temperature °C	Exhaust temperature °C	FC gram/h	FC MJ/h	NOx PPM	HC PPM	CO %	O ₂ %	CO ₂ %
124	284	1091	22.10	12	184	0.20	15.0	4.4
105	n.a.	1157	23.44	7	270	0.25	14.1	4.5
80	n.a.	1215	24.61	3	486	0.32	14.2	4.3

Discussion of Results

The obtained fuel efficiency based on equivalent energy amount was like neat DME at 125°C inlet temperature. A

clear trend was seen when air inlet temperature was reduced. The fuel efficiency was reduced and at the same time CO and HC were increased as a result of the deteriorated combustion. It is seen that NOx values are reduced to a very low level. The actual emission at inlet temperature 125°C compared to diesel fuel operation at the same conditions was reduced by a factor of 32.

Example 4

Diesel Fuel Composition (Fuel No. 4)

DME/methanol/water composition = 60/20/20 wt/wt%.

According to the chemical equilibrium calculations shown in Fig. 1 concentrations close to 60% DME, 20% methanol and 20% water all by mass are obtained at 400°C dehydration temperature and 20 bar. A fuel mixture having this composition was prepared and tested according to the test procedure described above. The diesel engine was started on neat DME. Load on the engine was set to 40% by means of the electrical heater (1Kw). Air inlet was heated to 125°C and stabilised. The 3-way valve was switched to fuel mixture No. 4. It was observed that the engine kept running also on this fuel composition. The combustion was even at the 125°C inlet temperature. The fuel consumption was not measured in this data series and only O₂, CO₂ and CO in the exhaust gas were measured. However, the results of combustion behaviour at various inlet temperatures were achieved. Results are given below in Table 6.

The air inlet temperature were reduced to 106°C. The engine operation were uneven and misfiring were observed. Further reduction of air inlet temperature to 77°C resulted in a

definitely more uneven operation and at 60°C the engine stopped.

Table 6

Inlet Air tempera- ture °C	Exhaust tempera- ture °C	FC gram/h	FC MJ/h	NOx PPM	HC PPM	CO %	O ₂ %	CO ₂ %
125	341	n.a.	n.a.	n.a.	371	0.27	13.6	5.5
106	357	n.a.	n.a.	n.a.	701	0.31	13.6	5.6
77	377	n.a.	n.a.	n.a.	1705	0.33	13.1	5.8
60	Engine stop							

Discussion of Results

The results indicate that inlet air temperature of 125°C with this composition is necessary to obtain satisfactory combustion.

Example 5

Diesel Fuel Composition (Fuel No. 5)

DME/methanol/water/DEE composition = 50/20/20/10 wt/wt%. According to the chemical equilibrium calculations, concentrations of close to 60% DME plus 20% methanol and 20% water all by mass are obtained at 400°C dehydration temperature and 20 bar if neat methanol is used as feed for the dehydration reactor. To simulate dehydration at this temperature and to simulate a methanol feed fuel containing ethanol for the dehydration reactor 10% of the DME were replaced by diethylether (DEE), which is the ether formed by dehydration of ethanol. Naturally, a specific composition of a dehydrated product would have a content of unconverted

ethanol too. However, the intention of testing this composition were to substantiate the combustion properties of fuel compositions generated from an ethanol containing methanol feed fuel for the dehydration reactor by preparing and testing this fuel No. 5.

A fuel mixture having this composition was prepared, placed on the balance and tested according to the test procedure described above. The diesel engine was again started on neat DME. Load was set to 40% by means of the electrical heater (1kW). Air inlet was heated to 127°C and stabilised for 5 minutes. The 3-way valve was switched to fuel mixture No. 5. The engine kept running on the specified fuel composition. Fuel consumption and emissions were recorded at three air inlet temperature levels as listed in Table 7.

Table 7

Inlet Air tempera- ture °C	Exhaust tempera- ture °C	FC gram/h	FC MJ/h	NOx PPM	HC PPM	CO %	O ₂ %	CO ₂ %
127	337	1080	23.53	10	278	0.25	13.4	4.9
101	346	1176	25.63	8	513	0.29	13.3	4.9
70	360	1275	27.78	9	1000	0.31	13.1	5.0

Discussion of Results

According to Table 7 results confirmed earlier observations; very low NOx formation was obtained with these aqueous fuel compositions. Good fuel efficiency at 127°C was obtained and deteriorated combustion associated with decreased efficiency at lower inlet temperatures.

Example 6**Diesel Fuel Composition (Fuel No. 6)**

DME/methanol/water composition = 48/4/48 wt/wt%.

5 The present fuel composition serves as example for the high water tolerance of fuel compositions used in diesel engines with preheated air. A total mapping of all possible fuel compositions is not meaningful. The present fuel composition serves as example of a fuel extremity regarding water content. A more meaningful definition of suitable fuel compositions obtained by dry methanol dehydration and by aqueous methanol dehydration is given in Fig. 1. It appears that fuel compositions obtained having a water content in between the two lines (A and B) are suitable fuels providing a minimum conversion of methanol providing enough ether to start ignition. In the present example, 48% DME were sufficient. However, the lowest acceptable limit of ether content in the fuel composition is associated with the specific engine and the operation conditions for the engine. A fuel mixture having the above-described composition was prepared, placed on the balance and tested according to the test procedure described above. The diesel engine was started on neat DME. Load on the engine was set to 40% by means of the electrical heater (1kW). Air inlet was heated to 125°C and stabilised for 5 minutes. The 3-way valve was switched to fuel mixture No. 5. The engine kept running on the specified fuel composition. Fuel consumption and emissions were recorded at three air inlet temperature levels as listed in Table 8.

Table 8

Inlet Air tempera- ture °C	Exhaust tempera- ture °C	FC gram/h	FC MJ/h	NOx PPM	HC PPM	CO %	O ₂ %	CO ₂ %
125	250	1674	24.49	7	239	0.20	14.4	4.6
98	248	1650	24.14	6	365	0.32	14.4	4.3
78	250	1920	28.09	8	650	0.41	14.4	4.2

Discussion of Results

- 5 It can be concluded that the ether ability to ignite even in mixtures with 48% water is satisfactory. However, fuel efficiency is reduced at these very high concentrations. The NOx formation seems to be much suppressed at this low exhaust gas temperature and high partial pressure of water.

Table 9
Summary of Achieved Results

Fuel composition DME/MeOH/H ₂ O/DEE wt/wt%	Air inlet tempera- ture °C	Fuel Consump- tion at 40% load MJ/h	NO _x PPM	HC PPM	CO %
Diesel	10	22.63	388	31	0.063
DME	10	22.74	119	163	0.073
DME	124	21.80	259	64	0.042
60/15/25/0	120	22.10	12	184	0.20
	100	23.44	7	270	0.25
	80	24.60	3	486	0.32
60/20/20/0	125	-	-	371	0.27
	106	-	-	701	0.31
	77	-	-	1705	0.33
50/20/20/10	127	23.53	10	278	0.25
	101	25.63	8	513	0.29
	70	27.78	9	1000	0.31
48/4/48/0	125	24.49	7	239	0.20
	98	24.14	6	365	0.32
	78	28.09	8	650	0.41

International Patent Application No. PCT/EP00/05275

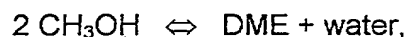
Haldor Topsøe A/S

PCT 1182-00957

May 18, 2001

NEW CLAIMS 1 TO 5

1. A process of preparing an oxygenated diesel fuel composition sustainable for use in compression ignition internal combustion engines, comprising methanol, dimethyl ether and water, comprising the step of converting methanol containing up to 20% w/w of water and up to 20% w/w of ethanol or higher alcohol in a catalytic dehydration reaction, the methanol being converted to dimethyl ether according to the reaction scheme:



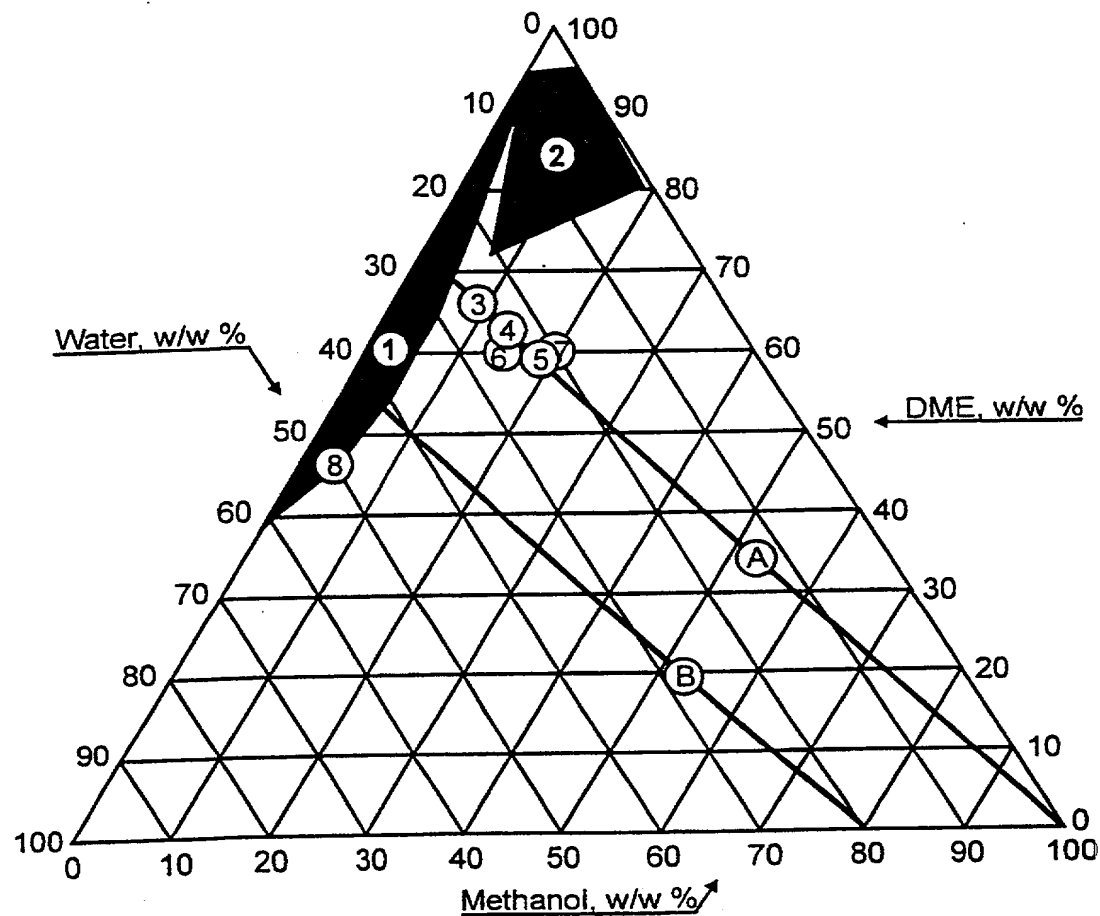
using a catalytic converter on board of a vehicle, wherein the dehydration temperature is between 200°C and 450°C and wherein the pressure is between 10 and 400 bar.

2. A method of operating a compression ignition engine on a fuel obtainable by the process of claim 1, by injecting the fuel into the combustion chamber of the engine and combusting the fuel with air, wherein the concentration of methanol is between 5 and 50% w/w and wherein the air for combustion is preheated to a temperature of at least 60°C.
3. The method of claim 2, wherein the combustion air is preheated to a temperature of at least 100°C.
4. The method of claim 2, wherein the combustion air is preheated by exchange with the exhaust gas.
5. The method of claim 2 applied to vehicles, ships, trains or in stationary diesel engines for power and heat supply.

1/2

Fig. 1

Trapeze diagram showing physical conditions for DME, methanol and water,
Equilibrium conditions, tested fuel compositions and various patented areas



① Two liquid phases

② HTAS/Amoco Patented

On Board MeOH to DME conversion :

③ Equilibrium 200°C, 20 bar

④ Equilibrium 300°C, 20 bar

⑤ Equilibrium 400°C, 20 bar

⑥ Test Fuel # 3

⑦ Test Fuel # 4

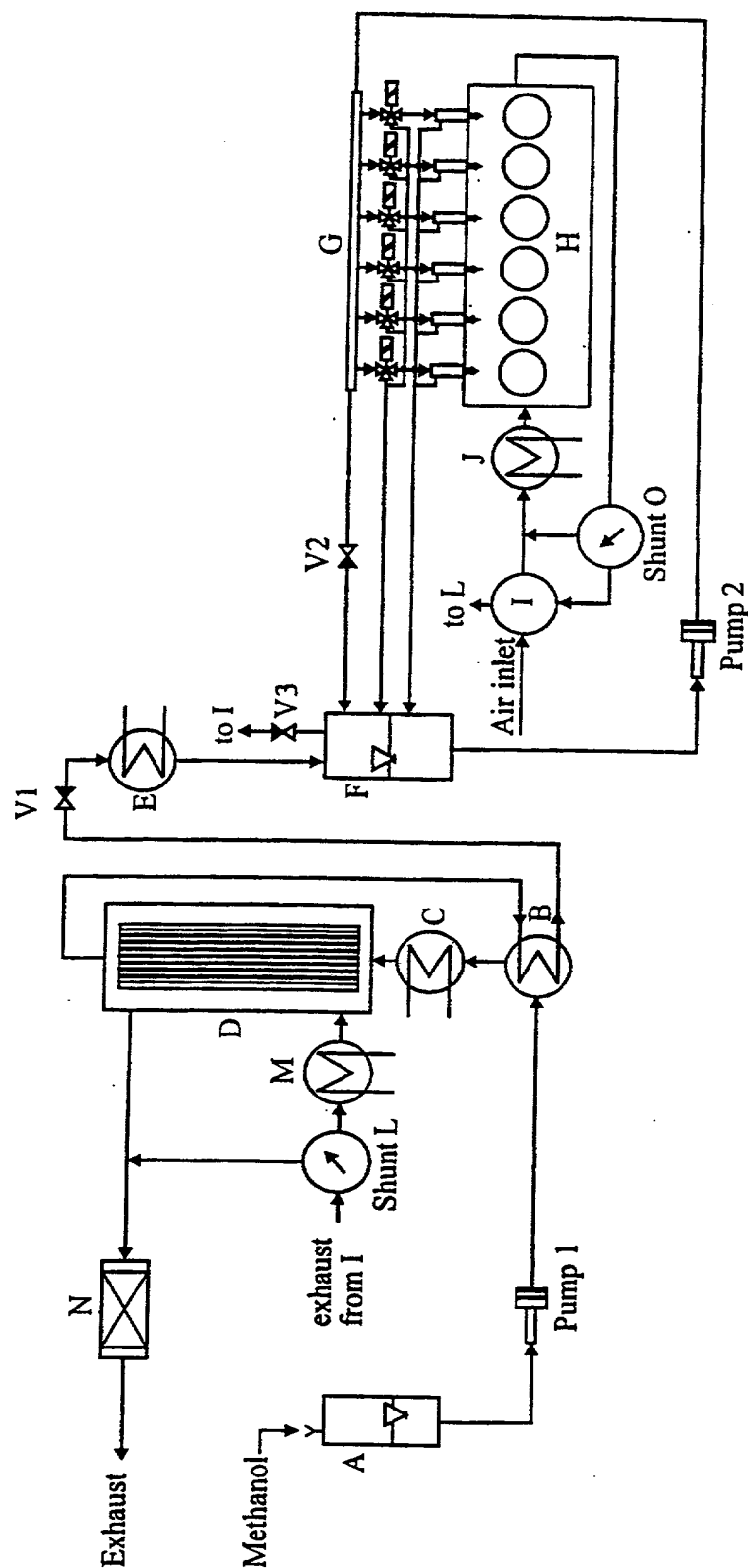
⑧ Test Fuel # 6

Ⓐ - MeOH Dehydration Fuel (Dry)

Ⓑ - MeOH Dehydration Fuel (Aqueous)

FIG. 2

Simplified Flow Sheet for Methanol to DME Fuel Processing Unit for Mobil or Stationary Diesel Engines



APR 17 2002

Declaration for Patent Application English Language Declaration

Attorney Docket No.	G3781.0005/P005
First Name Inventor	Svend-Erik Mikkelsen
COMPLETE IF KNOWN:	
Application No.	10/019,485
Filing Date	December 31, 2001
Group Art Unit	N/A
Examiner	Not Yet Assigned

☐ Submitted with initial filing

☒ Submitted after initial filing (surcharge required 37 CFR 1.16(e))

As a below named inventor, I hereby declare that:

My residence, mailing address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

CONTINUOUS DEHYDRATION OF ALCOHOL TO ETHER AND WATER USED AS FUEL FOR DIESEL ENGINES

the specification of which

☐ is attached hereto
OR
☒ was filed on 06/07/00 (Int'l. Filing Date) 12/31/2001 (U.S. Filing Date) PCT/EP00/05275 (PCT)
as United States Application No. or PCT International Application No. 10/019,485 (U.S.)
and was amended on _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

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I hereby claim foreign priority benefits under 35 U.S.C. 119(a)-(d) or (f), or 365(b) of any foreign applications(s) for patent, inventor's or plant breeder's rights certificate(s), or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent, inventor's or plant breeder's right certificate(s), or any PCT international application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application(s)

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				YES	NO
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PCT/EP00/05275 (Number)	PCT (Country)	June 7, 2000 (Filing Date)	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>
 (Number)	 (Country)	 (Filing Date)	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

☐ Additional prior foreign applications are listed on a supplemental data sheet attached hereto.

2047455-041702

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	Filing Date	December 31, 2001
	First Named Inventor	Svend-Erik Mikkelsen
	Title	CONTINUOUS DEHYDRATION OF ALCOHOL TO ETHER, etc.
	Group Art Unit	N/A
	Examiner Name	Not Yet Assigned
	Attorney Docket No.	G3781.0005/P005

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James W. Brady, Jr.	32,115	John C. Luce	34,378
Jon D. Grossman	32,699	Peter McGee	35,947
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Assignee of record of the entire interest. See 37 CFR 3.71.

Statement under 37 CFR 3.73(b) is enclosed. (Form PTO/SB/96).

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Name	Svend-Erik Mikkelsen
Signature	
Date	February 14, 2002

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	Group Art Unit	N/A
	Examiner Name	Not Yet Assigned
	Attorney Docket No.	G3781.0005/P005

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☒ Applicant/Inventor.

☐ Assignee of record of the entire interest. See 37 CFR 3.71.
Statement under 37 CFR 3.73(b) is enclosed. (Form PTO/SB/96).

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Date	February 14, 2002

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	Filing Date	December 31, 2001
	First Named Inventor	Svend-Erik Mikkelsen
	Title	CONTINUOUS DEHYDRATION OF ALCOHOL TO ETHER, etc.
	Group Art Unit	N/A
	Examiner Name	Not Yet Assigned
	Attorney Docket No.	G3781.0005/P005

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Mark J. Thronson	33,082	William E. Powell, III	39,803

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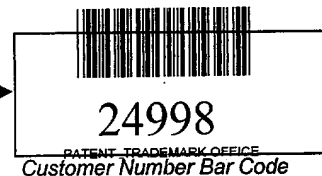
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John B. Hansen, Senior Scientist

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February 14, 2002

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Gianni Minutoli	41,198		
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